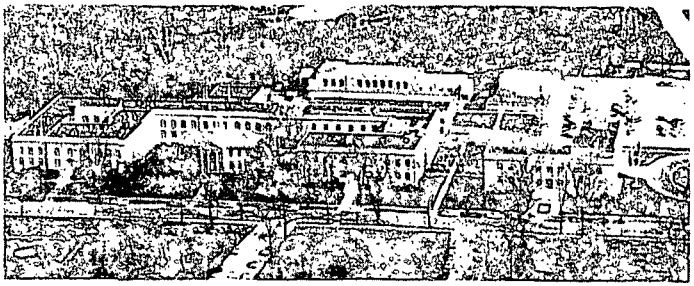

THE INSTITUTE OF PAPER CHEMISTRY, APPLETON, WISCONSIN

DEVELOPMENT OF A MANUFACTURING PROCEDURE FOR LOW-LITHIUM, LOW-URANIUM CONTENT FILTER PAPER

Project 3101

Report Six

A Status Report

to

DEPARTMENT OF THE AIR FORCE
1155th TECHNICAL OPERATIONS SQUADRON (HQ. COMD.)
McCLELLAN AFB, CALIFORNIA

July 10, 1973

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

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SUMMARY

Stacks of Hercules PS-57 pulp and IPC-1478 paper (without Kronisol) were leached with hydrofluoric acid and ammonium carbonate. By repeating the leaching and drying process once or twice on the pulp, lithium contents of < 0.4 ng. Li/g. were obtained consistently. All samples of IPC-1478 paper had been leached only once and retained 0.6-1.0 ng. Li/g. Uranium contents of IPC-1478 paper and most samples of pulp were consistently < 0.3 ng. U/g.

Work is in progress to test the repeated leaching and drying cycles on both pulp and paper to achieve the low levels of lithium required. Also, a quantity of purified pulp will be made into handsheets to test the effect of low consistencies on the retention of lithium and uranium.

INTRODUCTION

Work was continued in the use of hydrofluoric acid and ammonium carbonate to remove the lithium and uranium from IPC-1478 paper and from a cotton linter pulp, Hercules PS-57. As indicated by the results obtained previously, the percolation technique was efficient and reproducible and was used in the current work.

On May 22-23, Captains Dave Paquette, James Wright, and Frank Grosso visited the Institute to review the status of the work on Project 3101. Many aspects of the work were evaluated, and high priority was given to plans for making paper, as handsheets, from highly purified pulp.

DISCUSSION

The results reported in Project Report Five (1) indicated that the Hercules pulp, PS-57, could be purified to lithium contents of < 0.4 ng./g., but that IPC-1478 paper retained amounts of lithium in excess of the goal. Guided by these results, circles of IPC-1478 paper were placed in stacks of pulp and leached with hydrofluoric acid to achieve maximum exposure to the leaching reagents and wash water, and a minimum of contamination by solution-borne and air-borne materials. The results are summarized in Table I.

By inspection of the analytical data for the controls of the IPC-1478 paper (Sample No. 013-5501) and of the purified paper samples (No. 3101-142, 143, and 146), the leaching process removed approximately 90% of the lithium present in the original paper. However, Samples 3101-137 and 138 appeared to retain considerably more lithium than the others. In all cases of the IPC-1478 paper, the sample was consumed in obtaining the data as shown, and confirmation of the analysis is not possible.

The uranium contents, 0.065-0.165 ng. U/g., of these purified papers were below the goal of < 0.3 ng./g. As expected, the use of ammonium carbonate (Samples 3101-138, 145, and 146) appeared to favor the removal of uranium over samples (3101-137, 139, 140, and 141) leached with hydrofluoric acid, only, but the differences were small.

Pulp samples (No. 3101-119 to 127, Report Five) which had been leached once with hydrofluoric acid and ammonium carbonate retained 0.3 to 0.7 ng. Li/g. of pulp, but the samples (3101-139, 141, 144, 145 and 147) which had been leached

TABLE I

IPC-1478 PAPER AND HERCULES COTTON LINTER PULP
LEACHED WITH AMMONIUM CARBONATE AND
HYDROFLUORIC ACID[a]

Sample No.	Amount and Form of Pulp or Paper	Leaching Reagent(s)		Lithium, ng./g.	Uranium β/5	Uranium ng./g.
3101-137	IPC-1478 paper (Sample PM 7061A, Roll 2, Buckeye Linter, Double Pass), stacked with disks of Hercules pulp, PS-57	2M hydrofluoric acid; 0.5M hydrofluoric acid; water	A	1.30	127	0.148
			B	0.92	126	0.129
138	IPC-1478 paper (same as 3101-137)	0.5M Ammonium carbonate; water; 2M hydrofluoric acid; water	A	4.12[c]	132	0.065
			B	NR	126	0.074
139 (upper section)	Pulp disks, Hercules PS-57, previously leached	Stack of pulp disks and paper; leached with 2M hydrofluoric acid; water	A	0.375	116	0.189
			B	0.369	120	0.213
140	Pulp, reduced with sodium borohydride			0.311	135	1.55
141 (lower section)	Pulp disks (same as 3101-139)		A	0.418	118	0.336
			B	0.351	118	0.328
142	IPC-1478 paper (Sample PM 7062A, Roll 1, Hercules[e] Linters)	2M Hydrofluoric acid; water		0.666	128	0.132
143	IPC-1478 paper (same as 3101-142)	0.5M Ammonium carbonate; water; 2M hydrofluoric acid; water		0.838	133	0.165
144	Pulp disks from stack with 3101-143		A	0.315	136	0.223
			B	0.288	134	0.081
145	Pulp disks, previously extracted (as in 3101-125 to -127, see Report Five)	0.5M Ammonium carbonate; water; 2M hydrofluoric acid; water	A	0.144[d]	136	0.064
			B	0.105	135	0.096
146	IPC-1478 paper (Sample PM 7062A, Roll 1) stacked with previously-extracted pulp (3101-145)	0.5M Ammonium carbonate; water; 2M hydrofluoric acid; water		0.593[d]	132	0.104
147	Pulp disks from 3101-146		A	0.206[d]	141	0.277
			B	0.186	137	0.092
013-5501[b]	PM-7061A, Roll 2, Buckeye Linters	Control	A	7.80	131	0.800
			B	7.51	137	0.730
	PM-7062A, Roll 1, Hercules SFP	Control	C	6.44	127	1.18
			D	6.42	128	1.26
3101-001	Pulp, Hercules, PS-57 (see Project 3101, Report One)	Control		2.37	116	1.26

[a] All analytical data were obtained at the McClellan AFB, MCL-C, and were transmitted in a letter dated June 15, 1973, from Captain Frank Grosso to E. E. Dickey.

[b] Sample number assigned by McClellan AFB, MCL-C.

[c] Represents the average of three analyses.

[d] Represents the average of two analyses.

[e] The package in which this sample was received from McClellan AFB, MCL-C, was labeled "Buckeye," but the paper is believed to have been made from a Hercules pulp.

once or twice more, were consistently below 0.4 ng. Li/g. The uranium contents of the pulp samples were generally < 0.2 ng. U/g., especially when ammonium carbonate was used in the process.

FUTURE WORK

1. Experiments are underway to test the process of repeated leaching of IPC-1478 paper (no Kronisol added) with hydrofluoric acid. The paper will be processed in stacks of Hercules PS-57 pulp to secure maximum protection from water-borne and air-borne contamination.

2. A quantity of pulp will be purified by repeated leaching with hydrofluoric acid and will then be made into handsheets of approximately the same basis weight as IPC-1478 paper. In the first series of experiments, scrim will be omitted in order to study the pattern of lithium retention by the purified pulp in the papermaking process.

EXPERIMENTAL

3101-137. IPC-1478 PAPER LEACHED WITH HYDROFLUORIC ACID

Four circles of IPC-1478 paper, 12.5 cm. in diameter [PM 7061A, Roll 2, Buckeye Linters, Double Pass] were stacked with disks (octagons 15 cm. in diameter) so that each circle of paper was sandwiched between pulp disks. Five additional pulp disks were placed on top and two on the bottom of the stack. The stack was weighted and leached by gravity with 0.6 liter of 2.0M hydrofluoric acid then 0.8 liter of 0.5M hydrofluoric acid, and finally with 1.2 liters of water. Each solution was applied in small portions so that the exposure of the stack to each step was about 60 minutes. The stack was pressed and the circles were dried between pulp disks.

3101-138. IPC-1478 PAPER LEACHED WITH AMMONIUM CARBONATE AND HYDROFLUORIC ACID

Six circles of the IPC-1478 paper [same lot as in 3101-137] were sandwiched between pulp disks as previously described. The stack was leached with 1.0 liter of 0.5M ammonium carbonate, 0.8 liter of water, 0.7 liter of 2M hydrofluoric acid, and finally with 1.5 liters of water. The stack was in contact with each solution for sixty minutes, the final water-wash was completed after standing overnight, and the stack was pressed and dried.

3101-139 to -141. PULP TREATED WITH SODIUM BOROHYDRIDE AND LEACHED WITH HYDROFLUORIC ACID

An amount of 10 g. of pulp which had been treated with sodium borohydride was suspended in water and formed into a mat 12.5 cm. in diameter. The mat was placed between the 15th and 16th disks in a stack of 22 disks of pulp.

The stack was weighted, leached with 0.7 liter of 2M hydrofluoric acid followed by 0.5 liter 0.5M hydrofluoric acid, and finally with water until the effluent was neutral to Congo Red paper. The stack was pressed under a rubber dam, the disks 11-14, 17-20, and 15-16 with the treated mat were dried as sets. When dry, disks 12 and 13, the treated mat, and disks 18 and 19 were submitted for analysis as 3101-139, -140, and -141, respectively.

3101-142 to -144. IPC-1478 PAPER LEACHED WITH HYDROFLUORIC ACID AND AMMONIUM CARBONATE

Four circles (12.5 cm.) of IPC-1478 paper [PM 7062A, Roll 1, Hercules[a] Linters] in sets of two each were placed in a stack of previously processed (3101-119) disks of pulp. The stack consisted of 22 disks (200 g.) with one set of the IPC-1478 paper placed between the 16th and 17th disks and the other set between the 18th and 19th disks, respectively. The stack was weighted and was leached with 0.6 liter of 2M hydrofluoric acid followed by 0.7 liter of water. The stack was pressed in the usual way and dried as No. 3101-142.

The experiment was repeated except that the stack was leached with 0.5 liter of 0.5M ammonium carbonate and water before the extraction with hydrofluoric acid. The sample was dried as No. 3101-143. Three sets of three each of the pulp disks stacked above the IPC-1478 paper were dried and the middle disks of each set were combined as pulp Sample 3101-144.

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[a]The package in which this sample was received from McClellan AFB, MCL-C, was labeled "Buckeye," but the paper is believed to have been made from a Hercules pulp.

3101-145. PULP LEACHED REPEATEDLY WITH HYDROFLUORIC ACID AND AMMONIUM CARBONATE

An amount of 200 g. of pulp disks had been leached with 0.5M hydrofluoric acid, washed with water, and dried (see 3101-125 to -127). The stack was weighted and was leached with 0.5 liter of 0.5M ammonium carbonate, washed with 0.7 liter of water, leached with 0.5 liter of 2M hydrofluoric acid, washed with water (1.5 liters) until neutral to Congo Red paper, and dried in sets of three. The middle disks were collected for analysis.

3101-146 and -147. IPC-1478 PAPER LEACHED WITH AMMONIUM CARBONATE AND HYDROFLUORIC ACID

Six circles (12.5 cm. in diameter) of IPC-1478 paper [PM 7062A, Roll 1] were placed in sets of two each in a stack of pulp disks (same as 3101-145) which had previously been extracted. The stack was then leached with 0.5 liter of 0.5M ammonium carbonate, 0.5 liter of water, 1.0 liter of 2M hydrofluoric acid, 1.5 liters of wa er, pressed, and dried. The IPC-1478 paper (3101-146) was dried between pulp disks. Four pulp disks from the section of the stack above the IPC-1478 paper were submitted as 3101-147.

LITERATURE CITED

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